A STUDY ON ANALYSIS AND PREDICTION OF EROSION RESPONSE OF PLASMA SPRAYED ALUMINA COATINGS

A Project Report Submitted in Partial Fulfillment of the Requirements for the Degree of

B. Tech.

(Mechanical Engineering)

By

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ROURKELA

MAY, 2012

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C E R T I F I C A T E

This is to certify that the work in this project report entitled "**A STUDY ON ANALYSIS AND PREDICTION OF EROSION RESPONSE OF PLASMA SPRAYED ALUMINA COATINGS"** by **NISHAN DAS** has been carried out under my supervision in partial fulfillment of the requirements for the degree of **Bachelor of Technology** in Mechanical Engineering during session 20011- 2012 in the Department of Mechanical Engineering, National Institute of Technology, Rourkela.

To the best of my knowledge, this work has not been submitted to any other University/Institute for the award of any degree or diploma.

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A C K N O W L E D G E M E N T

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Abstract

This work presents successful implementation of Taguchi experimental design integrated with artificial neural networks (ANN) to develop a robust and efficient method of analyzing and predicting the erosion wear response of a new class of metal-glass coatings prepared by plasma spraying. Plasma spray technology utilizes the exotic properties of the plasma medium to effect physical, chemical or metallurgical reactions to produce new materials or impart new functional properties to conventional materials. Alumina (Al_2O_3)are preferred as the coating material over irregular ones due to low surface area to volume ratio, high density, free flowing ability and close sizing etc. Coatings of this alumina are deposited on mild steel substrates at various input power levels of the plasma torch. Erosion wear characteristics of these coatings are investigated following a plan of experiments based on the Taguchi technique, which is used to acquire the erosion test data in a controlled way. The study reveals that the impact velocity is the most significant among various factors influencing the wear rate of these coatings. An ANN model based on experimental data that performs self-learning by updating weightings is proposed in this work. It takes into account training and test procedure to predict the erosion performance under different erosive wear conditions. This technique helps in saving time and resources for a large number of experimental trials and successfully predicts the wear rate of the coatings both within and beyond the experimental domain.

Keywords: Plasma spraying, alumina, Erosion Wear; Taguchi Technique; ANN

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Chapter 1

Introduction

Chapter 1

INTRODUCTION

Surface modification is a generic term now applied to a large field of diverse technologies that can be gainfully harnessed to achieve increased reliability and enhanced performance of industrial components. The incessant quest for higher efficiency and productivity across the entire spectrum of manufacturing and engineering industries has ensured that most modern-day components are subjected to increasingly harsh environments during routine operation. Critical industrial components are therefore, prone to more rapid degradation as the parts fail to withstand the rigors of aggressive operating conditions and this has been taking a heavy toll of industry's economy. In an overwhelmingly large number of cases, the accelerated deterioration of parts and their eventual failure have been traced to material damage brought about by hostile environments and also by high relative motion between mating surfaces, corrosive media, extreme temperatures and cyclic stresses. Simultaneously, research efforts focused on the development of new materials for fabrication are beginning to yield diminishing returns and it appears unlikely that any significant advances in terms of component performance and durability can be made only through development of new alloys.

As a result of the above, the concept of incorporating engineered surfaces capable of combating the accompanying degradation phenomena like wear, corrosion and fatigue to improve component performance, reliability and durability has gained increasing acceptance in recent years. The recognition that a vast majority of engineering components fail catastrophically in service through surface related phenomena has further fuelled this approach and has led to the development of the broad interdisciplinary area of *surface modifications*. Thus, a protective coating deposited to act as a barrier between the surfaces of the component and the aggressive environment that it is exposed to during operation is now globally acknowledged to be an attractive means to significantly reduce/suppress damage to the actual component by acting as the first line of defense.

Typically, these coatings are aimed at modifying the surface properties of critical components to provide enhanced resistance against deterioration due to mechanisms such as corrosion, oxidation, wear or failure under an excessive heat load. In recent years, considerable advances in the field of coating technology have accompanied the growing realization of the immense potential of surface engineering in the modern industrial world. Consequently, there are now available a number of methods for developing a wide variety of protective coatings [1].

The increasing utility and industrial adoption of surface engineering is a consequence of the significant recent advances in the field. Very rapid strides have been made on all fronts of science viz. processing, control, modeling, application developments etc. and this has made it an invaluable tool that is now being increasingly considered to be an integral part of component design. Surface modification today is best defined as 'the design of substrate and surface together as a system to give a cost effective performance enhancement, of which neither is capable on its own'. The development of a suitable high performance coating on a component fabricated using an appropriate high strength metal/alloy offers a promising method of meeting both the bulk and surface property requirements of virtually all imagined applications. The newer surfacing techniques, along with the traditional ones, are eminently suited to modify a wide range of engineering properties. The properties that can be modified by adopting the surface engineering approach include tribological, mechanical, thermo-mechanical, electro-chemical, optical, electrical, electronic, magnetic, acoustic and biocompatible properties.

Driven by technological need and fuelled by exciting possibilities, novel methods for applying coatings, improvements in existing methods and new applications have proliferated in recent years. Surface modification technologies have grown rapidly, both in terms of finding better solutions and in the number of technology variants available, to offer a wide range of quality and cost. The significant increase in the availability of coating process of wide ranging complexity that are capable of depositing a plethora of coatings and handling components of diverse geometry today, ensures that components of all imaginable shape and size can be coated economically. Existing surface treatment and coating processes fall under three broad categories:

1. **Overlay Coatings**: This category incorporates a very wide variety of coating processes wherein a material different from the bulk is deposited on the substrate. The coating is distinct from the substrate in the as-coated condition and there exists a clear boundary at the substrate-coating interface. The adhesion of the coating to the substrate is a major issue in this process.

2. **Diffusion Coatings**: Chemical interaction of the coating-forming element(s) with the substrate by diffusion is involved in this category. New elements are diffused into the substrate surface, usually at elevated temperatures so that the composition and properties of outer layers are changed as compared to those of the bulk.

3. **Thermal or Mechanical Modifications of Surfaces**: In this case, the existing metallurgy of the component surface is changed in the near-surface region either by thermal or mechanical means, usually to increase its hardness.

As far as deposition of overlay coating is concerned, there are many techniques available, e.g. electroplating, vapour depositions, thermal spraying etc. Of all these coating methods, the thermal spray technique has gained by far the most widespread industrial acceptance. It has gradually emerged as the most industrially useful method of developing a variety of coatings, to enhance the quality of new components as well as to reclaim worn/wrongly machined parts. The type of thermal spraying depends on the type of heat source employed and consequently flame spraying (FS), high velocity oxy-fuel spraying (HVOF), plasma spraying (PS) etc. come under the umbrella of thermal spraying.

Plasma spray coating is a thermal spraying process that combines particle melting, quenching and consolidation in a single operation. It utilizes the exotic properties of the plasma medium to impart new functional properties to both conventional as well as non-conventional materials. The process involves injection of powder particles (metallic, ceramic or cermet powders) into the plasma jet created by heating an inert gas in an electric arc confined within a water-cooled nozzle. The temperature at the core of the plasma jet is 10,000-15,000 K. The particles injected into the plasma jet undergo rapid melting and at the same time are accelerated. These molten droplets moving at high velocities (exceeding 100 m/sec) impact on the surface of the substrate forming adherent coating [1, 2]. The coating is incrementally built up by impact of successive particles by the process of flattening, cooling and solidification. By virtue of the high cooling rates, typically 105 to 106 K/sec, the resulting microstructures are fine-grained and homogeneous [3].

Plasma spraying also has certain additional advantages over other competing surface engineering techniques. By virtue of the high temperature and high enthalpy of the thermal plasma jet, any powder, which melts without decomposition or sublimation, can be coated keeping the substrate temperature as low as 50^{o}C. The coating process is fast and the thickness can go from a few tens of microns to a few mm. It is a continuous process and can be used to deposit ceramics, metals, alloys and composites. Plasma spraying is extensively used in hi-tech industries like aerospace, nuclear energy as well as conventional industries like textiles, chemicals, plastics and paper mainly as wear resistant coatings in crucial components. It is a very large industry with applications in corrosion, abrasion and temperature resistant coatings and in the production of monolithic shapes [2]. This process can be applied to coat on variety of substrates of complicated shape and size using metallic, ceramic and/or polymeric consumables. The production rate of the process is very high and the coating adhesion is also adequate. It has therefore a very wide range of applicability, e.g., as thermal barrier coatings, wear resistant coatings etc. Thermal barrier coatings are provided to protect the base material in case of internal combustion engines, gas turbines etc. at elevated temperatures. Zirconia (ZrO_2) is well accepted as a conventional thermal barrier coating material. Similarly, as the name suggests, wear resistant coatings are used to combat wear especially in cylinder liners, pistons, valves, spindles, textile mill rollers etc. Alumina (Al_2O_3), titania (TiO_2) and zirconia (ZrO_2) are considered as some of the conventional wear resistant coating materials [1]. These materials form good adherent coatings on metallic substrates and are used in various thermal as well as tribological applications.

Plasma sprayed hard ceramic coatings are applied as protective layers on various engineering and structural components which are often used in situations where erosive wear occurs. Due to the operational requirements in dusty environments, the study of the erosion characteristics of these coatings becomes highly relevant. A full understanding of the effects of all operating and material variables on the wear rate is necessary in order to undertake appropriate steps in the design of components and in the choice of coating materials to reduce/control wear. The subject of erosion wear of plasma spray coatings has not received substantial research attention in the past although there is an increasing utilization of ceramic coatings in aerospace, transportation and process industries, where they can be subjected to multiple solid or liquid particle impact. Examples of such applications involving material loss by erosion wear are pipe lines carrying pulverized coal dust, helicopter rotor blades, compressor blades, high speed vehicles and aircrafts operating in desert environments etc. Despite its high significance, solid particle erosion behaviour of coatings has remained a less studied area. Hence, this aspect is taken up in the present investigation for some plasma sprayed coatings.

Statistical methods have commonly been used for analysis, prediction and/or parametric optimization of a number of engineering processes. Such methods enable the user to define and study the effect of every single condition possible in an experiment where numerous factors are involved. Solid particle erosion is a complex wear phenomenon in which a number of control factors collectively determine the performance output (i.e. the erosion rate) and there is enormous scope in it for the implementation of appropriate statistical techniques for parametric appraisal. But unfortunately, such studies have not been adequately reported so far. The present research work addresses to this aspect by adopting a statistical approach called Taguchi experimental design. This technique provides a simple, systematic and efficient methodology for the optimization of the control factors.

In the present investigation, a qualitative analysis of the experimental results with regard to erosion response of alumina based coatings using statistical techniques is presented. The analysis is aimed at identifying the operating variables/factors significantly influencing the erosion rate of coatings. Like any experimental investigation, erosion trials on plasma sprayed coatings also demand substantial amount of time, energy and materials. Hence, there is a need for a prediction tool to supplement the experiments. In the present study, a model based on artificial neural network (ANN) is implemented to predict the erosion rate of the coatings subjected to different operating conditions. ANN is an information processing paradigm that is inspired by the way that biological nervous systems process information. It is composed of a large number of interconnected processing elements (neurons) working in unison. Neural computation is used in the present work since solid particle erosion is a complex process that has many variables with multilateral interactions.

Against this background, the present research work is undertaken to study the development, characterization and erosion wear response of alumina based plasma sprayed coatings. The solid particle erosion characteristics of these coatings have been studied experimentally. The specific objectives of this work are clearly outlined in the next chapter.

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Chapter 2
Literature Review

Chapter 2

LITERATURE REVIEW

The purpose of this literature review is to provide background information on the issues to be considered in this thesis and to emphasize the relevance of the present study. This treatise embraces various aspects of ceramic coatings with a special reference to their erosion wear characteristics.

2.2 On Solid Particle Erosion Wear of Materials

Solid Particle Erosion (SPE) is a typical erosive wear mode where particles strike against surfaces and promote material loss. During flight a particle carries momentum and kinetic energy, which can be dissipated during impact, due to its interaction with a target surface. It is to be noted that solid particle erosion is different from the other forms of erosion like liquid impact erosion, slurry erosion, cavitation erosion etc. Material removal due to solid particle erosion is a consequence of a series of essentially independent but similar impact events. Thus, the contact between the hard particles and the component surface is of a very short duration. From this point of view, erosion is completely different from the other closely related processes like sliding wear, abrasion, grinding and machining wherein the contact between the tool/abrasive and the work-piece/target is continuous.

In some cases SPE is a useful phenomenon, as in sand-blasting and high-speed abrasive water jet cutting, shot peening of rotating components, cutting of hard and brittle materials by abrasive jets and rock drilling [4-6], but it is a serious problem in many engineering systems, including steam and jet turbines, pipelines and valves used in slurry transportation of matter and fluidized bed combustion systems. Gas and steam turbines operate in environments where the ingestion of solid particles is inevitable. In industrial applications and power generation, such as coal-burning boilers, fluidized beds and gas turbines, solid particles are produced during the combustion of heavy oils, synthetic fuels, pulverized coal etc. and causes erosion [23, 24] leading to the damage of compressor gas path components, such as stator vanes or rotor blades, leading to gradual changes in their surface finish and geometry [25, 26]. Similarly, a helicopter operating in a sandy or dusty field will generate a dust cloud that will be ingested by the compressor resulting in a progressive metal loss from both the leading and trailing edges of the airfoils [27]. Erosion is thus expected whenever hard particles are entrained in a gas or liquid medium impinging on a solid at any significant velocity. In both cases, particles can be accelerated or decelerated and their directions of motion can be changed by the fluid.

Degradation of materials due to solid particle erosion, either at room temperature or at elevated temperature, is encountered in a large variety of engineering industries as illustrated in table 2.1.

Systems	**Components**
Chemical plant	Transport tubes carrying abrasive materials in an air stream[28-30]
Hydraulic mining machinery	Pumps and valves[31]
Propellant system	Rocket motors trail nozzle, gun barrel[32]
Combustion system	Burner nozzle, reheater, super heater and economizer tube banks[33, 34]
Fluidized bed combustion	Boiler heat exchanger tubes in bed tubes, tube banks and expander turbine[33, 35-37]
Coal gasification	Turbine, lock hopper valves[33]
Coal liquefaction	Valve to throttle the flow of product steam[33]
Aircraft engine	Compressor and turbine blades[38]
Helicopter engine	Rotor and gas turbine blades[20]

Table 2.1 Examples of components subjected to solid particle erosion

In order to minimize damage caused by erosive wear, many authors propose the use of surface coatings. Fluidized bed combustion boilers, turbines and engines are normally exposed to erosive environments and the erosion leads to many accidents [39-42]. The ceramic coatings are considered as powerful barriers against deterioration of machine parts exposed to particulate flow at high temperatures [7]. Ceramic coatings have great potential for many applications due to their good thermal protectiveness, high hardness and wear resistance among others. For example, the wear resistant coatings are widely used in textile industry to improve the life time of different thread guiding elements, guiding and distribution rollers, ridge thread brakes, distribution plates, driving and driven rollers, gullets, tension rollers and thread brake caps [8].

Applications of ceramic coatings produced by different deposition methods are increasingly used to extend the service life of mechanical components. This is because the coatings themselves have high hardness and chemical inertness and have excellent wear resistance, which makes it possible to protect the surface from erosive environments. Friction and wear are surface phenomena and are of high concern especially in industrial components resulting in huge economic losses and sometimes lead to catastrophic failure. Hence, it is of utmost importance to minimize their ill effects. Use of coatings would enhance the wear resistance as well as anti-friction resistance of the materials. In addition, coatings enable use of relatively cheaper materials for machine components. Recently, cermet coatings are used to further increase the erosion resistance through increasing the toughness of the coatings [9-13]. The erosion resistance of the coatings is influenced not only by the impact angle, particle velocity and environment temperature, but also is strongly dependent on the coating process. For the application of these materials to components, different techniques in the field of surface engineering can be considered. Some researchers made use of processes such as thermal spraying, sputtering, physical vapour deposition, chemical vapour deposition, detonation spraying and electro-spark detonation to obtain protective coatings against erosive wear. Out of all these surface modification techniques, however, the most widely reported one is thermal spraying.

2.4 On Plasma Spray Coating Technique

Plasma spray coating technique utilizes the exotic properties of the plasma medium to effect physical, chemical or metallurgical reactions to produce metallic and ceramic coatings for a variety of applications. Plasma is considered to be the fourth state of matter, consisting of a mixture of electrons, ions and neutral particles, although overall it is electrically neutral. The degree of ionization of a plasma is the proportion of atoms that have lost (or gained) electrons and in the case of thermal plasmas, this is controlled mostly by temperature. Plasma technology involves the creation of a sustained electrical arc by the passage of electric current through a gas in a process referred to as electrical breakdown. Because of the electrical resistivity across the system, significant heat is generated, which strips away electrons from the gas molecules resulting in an ionized gas stream or plasma. At about 2000^{o}C gas molecules dissociate into the atomic state and when the temperature is raised to about 3000^{o}C, gas molecules lose electrons and become ionized. In this state, gas has a liquid-like viscosity at atmospheric pressure and the free electric charges confer relatively high electrical conductivities that can approach those of metals [14].

Plasma spray coating is an economical and effective method applied to various machine parts to reduce the surface degradation. It is gaining importance in many critical areas of application due to the fact that it provides increased design flexibility and its high deposition rate, so that the parts made up from a combination of materials with widely differing physical and chemical properties could be employed. Plasma sprayed ceramic coatings have been widely used for industrial parts as well as in structural applications in order to improve resistances to wear, corrosion, oxidization, erosion and heat. In plasma spraying, a coated layer is formed on a substrate surface by spraying melted powders on to the substrate at a high speed using a high-temperature plasma heat source. The microstructure and properties of plasma sprayed coatings depend on the design of the plasma torch, the operating parameters including torch input power, plasma forming gases and flow rates, spray distance, feedstock composition, feed rate and injection parameters etc.

Plasma sprayed coatings are produced by introducing powder particles of the feedstock material into a plasma jet, which melts them and propels towards the substrate. The formation of a coating depends on the interaction between a droplet and the substrate or the previously deposited layers, i.e. spreading of a droplet, the formation of a splat (lamella) and its solidification. The difference in the degree of a splat flattening results in the difference in porosity and its shape as well as distribution and these factors could affect also the bonding between lamellae. A schematic diagram of plasma spray process is shown in the figure 2.1.

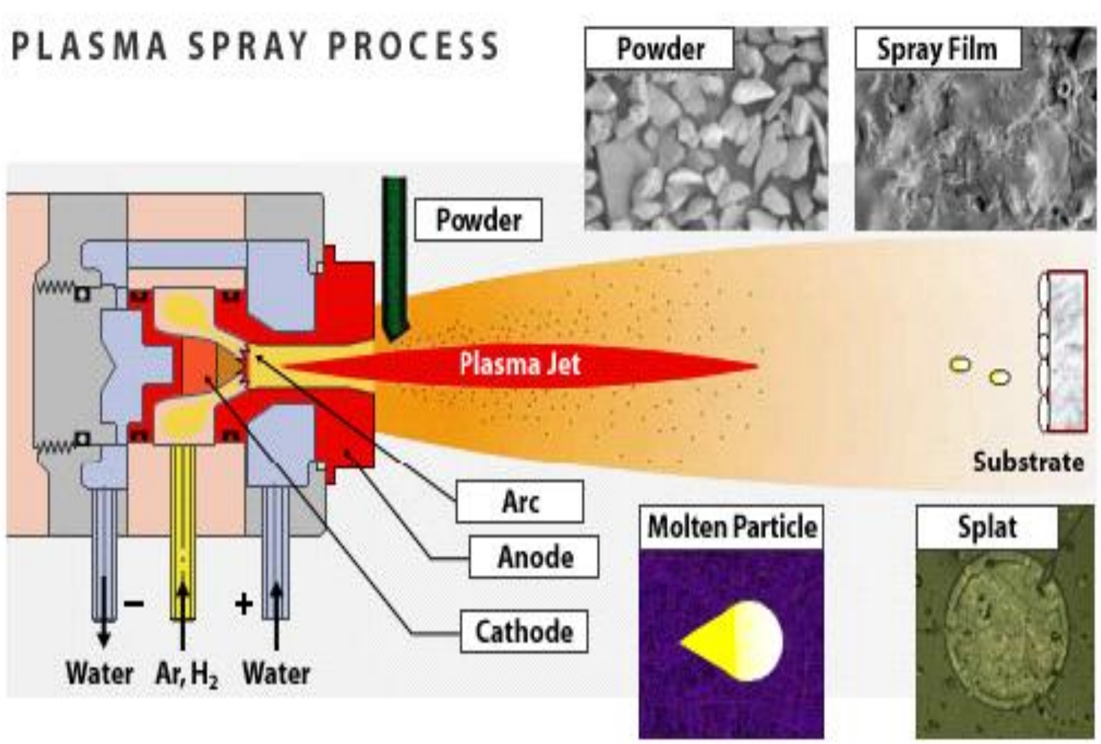

Figure 2.1 Conventional plasma spraying process

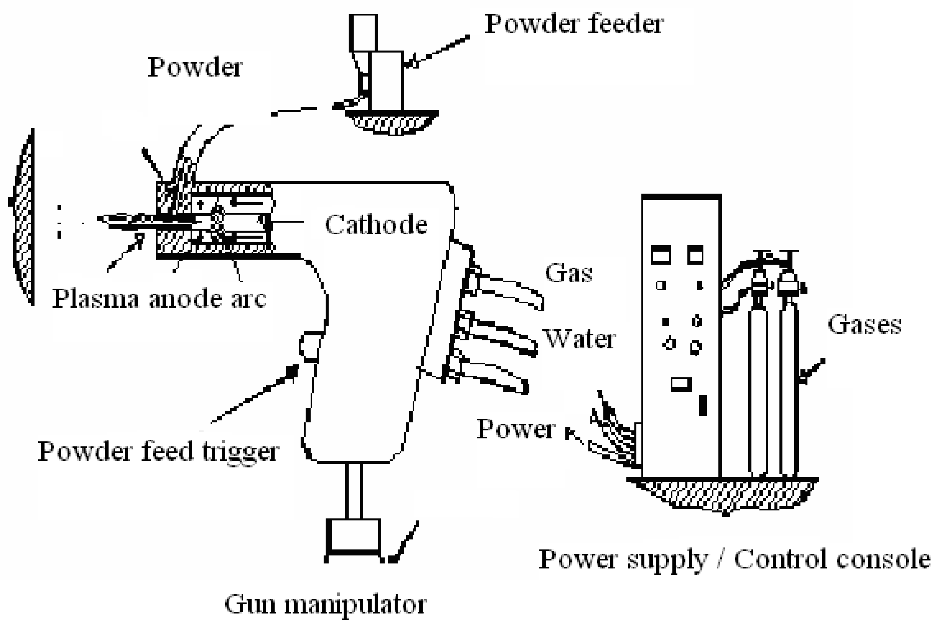

Figure 2.2 Arrangements for the plasma spraying

The general arrangement of a plasma spraying process is shown in figure 2.2. An arc is created between tungsten tipped copper cathode and an annular copper anode (both water cooled). Plasma generating gas is forced to pass through the annular space between the electrodes. While passing through the arc, the gas undergoes ionization in the high temperature environment resulting plasma. The ionization is achieved by collisions of electrons of the arc with the neutral molecules of the gas. The plasma protrudes out of the electrode encasement in the form of a flame. The consumable material, in the powdered form, is poured into the flame in metered quantity. The powders melt immediately and absorb the momentum of the expanding gas and rush towards the target to form a thin deposited layer. The next layer deposits onto the first one immediately after the deposition of first layer and thus the coating builds up layer by layer. The temperature in the plasma arc can be as high as 10,000^{o}C and it is capable of melting anything. Elaborate cooling arrangement is required to protect the plasmatron (i.e., the plasma generator) from excessive heating.

2.6 On Erosion Wear of Ceramic Coatings

Plasma sprayed coatings are used today as erosion resistant coatings in a wide variety of applications. Extensive research shows that the deposition parameters like energy input to the plasma and powder properties affect the porosity, splat size, phase composition, coating hardness etc. [15-18]. These in turn, have an influence on the erosion wear resistance of the coatings. Quantitative studies of the combined erosive effect of repeated impacts are very useful in predicting component lifetimes, in comparing the performance of materials and also in understanding the underlying damage mechanisms involved.

Resistance of engineering components encountering the attack of erosive environments during operation can be improved by applying ceramic coatings on their surfaces. Alonso et al. experimented with the production of plasma sprayed erosion-resistant coatings on carbon-fiber-epoxy composites and studied their erosion behaviour. The heat sensitivity of the composite substrate requires a specific spraying procedure in order to avoid its degradation. In addition, several bonding layers were tried to allow spraying of the protective coatings. Two different functional coatings; a cermet (WC-12 Co) and a ceramic oxide (Al_2O_3) were sprayed onto an aluminium-glass bonding layer. The microstructure and properties of these coatings were studied and their erosion characteristics were determined experimentally in an erosion-testing device.

Kulu et al. state that the solid particle erosion under extreme conditions (high hardness and strength of abrasives and materials to be ground, high velocity and pressure, cyclic impact load, elevated temperatures etc.) is a serious problem for industrial equipments, for example milling and mixing devices. It was shown that loading conditions (milling by collision, wear in the stream of hard particles, etc.) strongly influence the material behavior. As a result of erosive wear, the damage to the material depends on its structural characteristics and the properties associated with them. For brittle materials, brittle fracture is predominant, whereas in ductile ones the prevailing mechanisms are micro-cuts, platelet formation and/or low cycle fatigue.

Response of a material to SPE is a complex function of the physical properties of the target, the impacting particles and the erosive environment. Many erosion mechanisms have been proposed and supported by the experimental data from erosion tests and various models for the erosion of bulk metals, glass and ceramics have been proposed , usually considering different combinations of micro-cutting, plastic deformation, melting, fatigue and fracture mechanisms. According to Finnie and McFadden, there are four principal factors that influence the erosion behaviour of a material: the erodent particle velocity and size, the impact angle and the properties of the eroded material. Materials exhibit different types of erosion behaviour depending on the impact angle. In ductile materials, maximum erosion occurs at low-impact angles (generally between 15^o- 30^o with respect to the surface) as a result of shear deformation and micro-cutting. This mechanism of material removal is similar to the cutting action of machine tools. Brittle erosion occurs in hard materials (such as ceramics) as a result of crack network formation. Brittle materials erode most quickly at normal impact angle (90^o). However, transitions of the erosion behaviour of a material from one mechanism to another (ductile or brittle) were also reported in the past..

Branco et al. [19] examined room temperature solid particle erosion of zirconia and alumina based ceramic coatings, with different levels of porosity and varying microstructure and mechanical properties. The erosion tests were carried out by a stream of alumina particles with an average size of 50 µm at a velocity of 70 m/s, carried by an air jet with impingement angle of 90^o. The results of this study indicated that there is a strong relationship between the erosion rate and the coating porosity. Similarly, Mishra et al. investigated the erosion characteristics of plasma sprayed alumina-titania coating deposited on mild steel substrates. They reported that the erosion wear rate varied with the erodent dose, the angle of impact, erodent velocity, stand-off distance and also on the erodent size. This study revealed that alumina significantly improves the resistance of the coating to solid particle erosion. It also indicated that the peak erosion rate occurred at an impact angle of 90^o.

The exhaustive literature survey presented above on various issues related to this research work reveals that:

- Though much work has been reported on various wear characteristics of metals, alloys and homogeneous materials, comparatively less has been reported on the erosive wear performance of ceramic coatings and in fact very few studies have been found particularly on erosion of plasma sprayed ceramic coatings.
- Studies carried out worldwide on erosion behavior of plasma sprayed ceramic coatings have largely been experimental and use of statistical techniques in analyzing wear characteristics is rare.
- Taguchi method, in spite of being a simple, efficient and systematic approach to optimize designs for performance, quality and cost, is used only in a limited number of applications worldwide. Its implementation in parametric appraisal of wear processes has hardly been reported.

It is thus clear that the analysis of erosion wear response of plasma sprayed ceramic coatings has still remained a less studied area. It is felt that, a further study in this respect is needed particularly with the inclusion of a secondary metal/ceramic powder both in view of the scientific understanding and commercial importance.

In view of the above, the present work is undertaken to investigate the solid particle erosion wear characteristics of plasma sprayed alumina coatings under multiple impact conditions. The objectives of this work are outlined as follows:

1. Preparation of a series of plasma sprayed alumina coatings on metal substrates.
2. Study of erosion wear response of the prepared coatings and the parametric appraisal of the wear process using Taguchi experimental design.
3. Parametric analysis of wear rate of respective coatings based on Taguchi approach.
4. Implementation of an ANN based prediction model for estimation of erosion rate of the coatings under different test conditions.

Chapter Summary

This chapter has provided an outline of plasma spray technique and a report on the research carried out so far on the wear response of ceramic coatings. Finally it has clearly outlined the objectives of the present work.

The next chapter discusses the materials and methods, experimental planning and the Taguchi method.

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Chapter 3

Materials and Methods

Chapter 3

MATERIALS AND METHODS

This chapter deals with the details of the experimental procedures followed in this study. The coating procedure itself requires some basic preparations, i.e., shot blasting and cleaning of substrate surfaces to be coated. After plasma spraying, the coated samples have been subjected to solid particle erosion wear test The details of the test procedures are described below.

Preparation of Substrates

Mild steel is the most common form of steel because its price is relatively low while it provides material properties that are acceptable for many applications. Low carbon steel contains approximately 0.05–0.15% carbon and mild steel contains 0.16–0.29% carbon; making it malleable and ductile, but it cannot be hardened by heat treatment. Mild steel has a relatively low tensile strength, but it is cheap and malleable; surface hardness can be increased through carburizing. It is often used when large quantities of steel are needed, for example as structural steel. The density of mild steel is approximately 7.85 g/cm^3 (7850 kg/m^{3}or 0.284 lb/in^3) and the Young's modulus is 210 GPa (30,000,000 psi).

Coating Material (Alumina):

In this study, alumina is used as raw materials for coating deposition on mild steel substrates. Aluminium oxide is an amphoteric oxide with the chemical formula Al_2O_3. It is commonly referred to as alumina (α-alumina), or corundum in its crystalline form, as well as many other names, reflecting its widespread occurrence in nature and industry. Its most significant use is in the production of aluminum metal, although it is also used as an abrasive owing to its hardness and as a refractory material owing to its high melting point. There is also a cubic γ-alumina with important technical applications. Corundum is the most common naturally occurring crystalline form of aluminium oxide. Rubies and sapphires are gem-quality forms of corundum, which owe their characteristic colors to trace impurities. Rubies are given their characteristic deep red color and their laser qualities by traces of chromium. Sapphires come in different colors given by various other impurities, such as iron and titanium. Aluminium oxide is an electrical insulator but has a relatively high thermal conductivity (30 $Wm^{-1}K^{-1}$[1]) for a ceramic material. In its most commonly occurring crystalline form, called corundum or α-aluminium oxide, its hardness makes it suitable for use as an abrasive and as a component in cutting tools. The most common form of crystalline alumina is known as corundum. The oxygen ions nearly form a hexagonal close-packed structure with aluminium ions filling two-thirds of the octahedral interstices. Each Al^{3+} center is octahedral. In terms of its crystallography, corundum adopts a trigonal Bravais lattice with a space group of R-3c. The primitive cell contains two formula units of aluminium oxide. Alumina also exists in other phases, namely γ-, δ-, η-, θ-, and χ-aluminas. Each has a unique crystal structure and properties. The so-called β-alumina proved to be $NaAl_{11}O_{17}$

Commercially available mild steel plates have been chosen as the substrate material and they are cut into pieces of desired size. The specimens are rectangular in shape having dimensions of $50 \times 25 \times 3$ mm^3. The specimens are grit blasted at a pressure of 3 kg/cm^2 using alumina grits having size of around 60 µm. During sand blasting the average stand-off distance is kept constant at about 100 mm and the average roughness of the substrates obtained is 4.0-5.0 Ra. The grit blasted specimens are cleaned in an ultrasonic cleaning unit and the weight of each cleaned specimen is taken by using a precision electronic balance with ± 0.01 mg accuracy. Spraying on to these specimens is carried out immediately after weighing.

Plasma Spray Coating Deposition

In this investigation, coating depositions were carried out using a 80 kW plasma spray system (M/s Metallization make) at CSIR-IMMT, India. This is a typical atmospheric plasma spray system working in the non-transferred arc mode. The major sub-systems of the set up include the plasma spray torch, power supply, six-axis robot, mass flow controller, powder feeder, plasma gas supply, control console, cooling water and spray booth. A current regulated DC supply was used. A four stage centrifugal pump at a pressure of 10 kg/cm^2 supplied cooling water for the system. Argon and Helium taken from normal cylinders at an outlet pressure of 4 kg/cm^2 were used as plasma gas and carrier gas respectively. Plasma spray copper slag coatings are thus deposited over aluminium and mild steel substrates of dimensions 60×20×3 mm. Before deposition, all the substrates were sand blasted to get the required surface roughness. The powder feed rate was kept constant at about 20 gm/min. The operating parameters used in the experiments are presented in Table 3.1.

Table-3.1 Operating parameters used in the experiments

Parameter	Operating Range
Operating Power	10 – 25 kW
Current	400 - 600 A
Primary Plasmagen gas (Argon) flow rate	40 Lpm
Secondary Plasmagen gas (Helium) Flow rate	8 Lpm
Nozzle to substrate distance (Stand-off distance)	100 mm

The pictorial view of some typical coating samples made for this study is presented in the figure 3.4. The schematic sketch of a plasma sprayed coating showing the substrate, coating and the interfacial boundary is also illustrated in figure 3.6.

Figure 3.1 View of the atmospheric plasma spraying unit

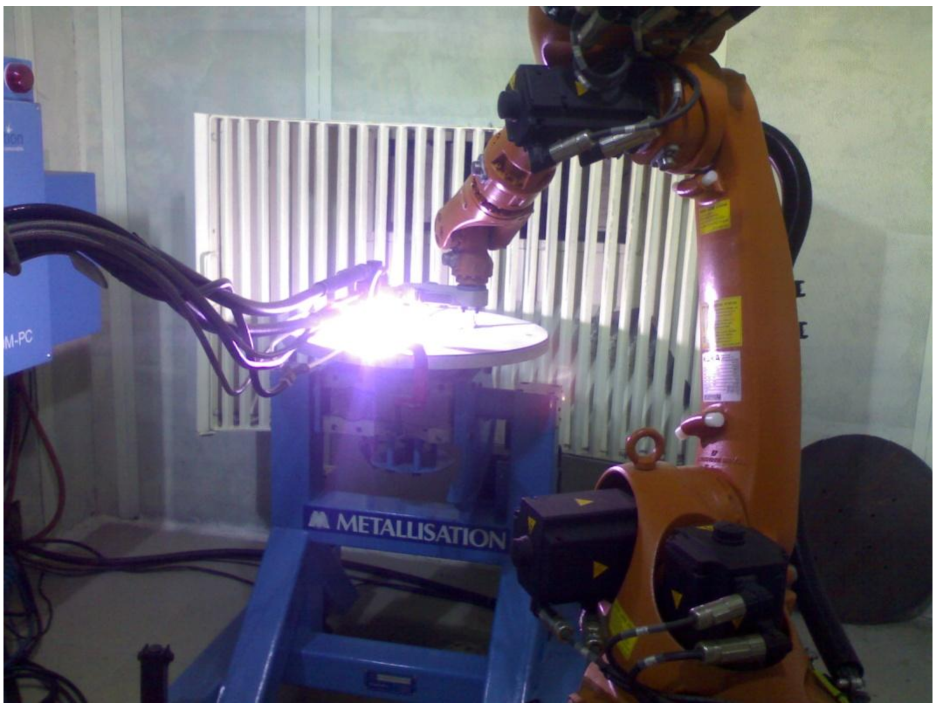

Figure 3.2 View of the coating deposition operation by plasma spraying

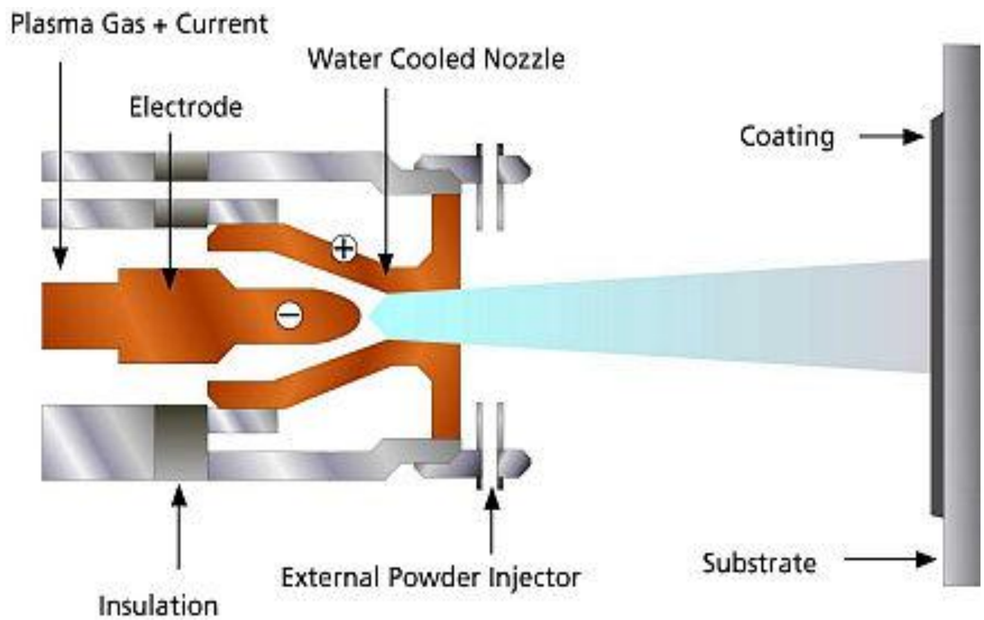

Figure 3.3 Schematic diagram of the plasma spraying process

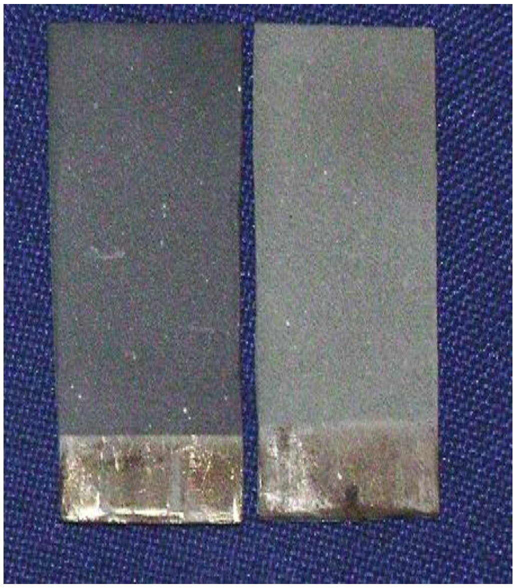

Figure 3.4 Pictorial view of plasma sprayed coating samples

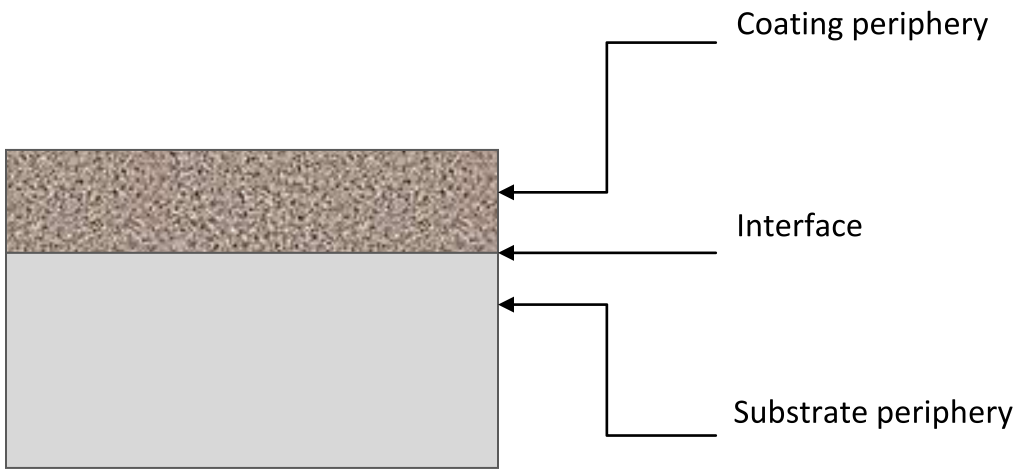

Figure 3.5 Schematic view of the plasma sprayed coatings

Solid Particle Erosion Wear of Coatings

Solid particle erosion (SPE) is usually simulated in laboratory by one of two methods. The 'sand blast' method, where particles are carried in an air flow and impacted onto a stationary target and the 'whirling arm' method, where the target is spun through a chamber of falling particles. In the present investigation, a self-made erosion apparatus of the 'sand blast' type is used. It is capable of creating highly reproducible erosive situations over a wide range of particle sizes, velocities, particle fluxes and incidence angles in order to generate adequate quantitative data on materials and to study the mechanisms of damage. The schematic diagram of the test rig and the photograph of the erosion set up are given in figures 3.6 and 3.7 respectively.

The air jet erosion test rig used in this work employs a 30 mm long nozzle of 3 mm bore. This nozzle size permits a wider range of particle types to be used in the course of testing, allowing better simulations of real erosion conditions. The mass flow rate is measured by conventional method. Particles are fed from a simple hopper under gravity into the groove. Velocity of impact is measured using the standard double disc method [331]. Some of the features of this test set up are:

- Vertical traverse for the nozzle: provides variable nozzle-to-target stand-off distance, which influences the size of the eroded area.
- Different nozzles may be accommodated: provides ability to change the particle plume dimensions and the velocity range.
- Large test chamber with sample mount (typical sample size 25×25 mm^2) that can be angled to the flow direction: by tilting the sample stage, the impingement angle of the particles can be changed in the range of 0^o-90^o.

In this work, room temperature solid particle erosion test on metal substrates coated with alumina is carried out at different impingement angles ranging from 30^o-90^o. Dry alumina particles of different average sizes are used as erodent. The test is conducted as per ASTM G76 standards.

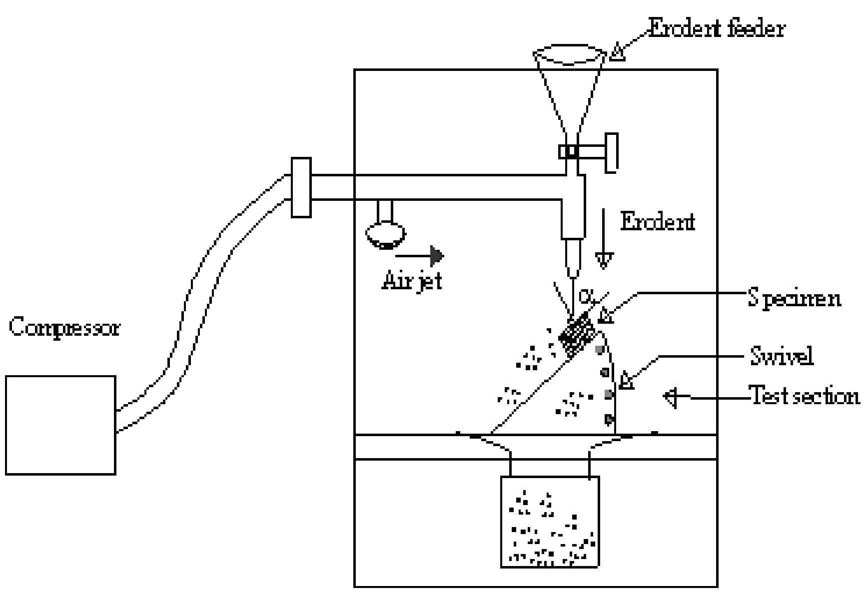

Figure 3.6 A schematic diagram of the solid particle erosion test rig

Figure 3.7 A pictorial view of the air jet type solid particle erosion test rig

Process Optimization and Taguchi Method

Statistical methods are commonly used to improve the quality of a product or process. Such methods enable the user to define and study the effect of every single condition possible in an experiment where numerous factors are involved. Solid particle erosion is such a process in which a number of control factors collectively determine the performance output i.e. the erosion rate. Hence, in the present work a statistical technique called Taguchi method is used to optimize the process parameters leading to minimum erosion of the plasma sprayed coatings under study. This part of the chapter presents the Taguchi experimental design methodology in detail.

Taguchi Experimental Design

Every single discipline in engineering has researchers carrying out experiments to observe and understand a certain process or to discover the interaction and effect of different variables on the output. From a scientific viewpoint, these experiments are either one or a series of tests to either confirm a hypothesis or to understand a process in further detail. In order to achieve a meaningful end result, several experiments are usually carried out. The experimenter needs to know the factors involved, the range these factors are varied between, the levels assigned to each factor as well as a method to calculate and quantify the response of each factor. This *one-factor-at-a-time* approach will provide the most favorable level for each factor but not the optimum combination of all the interacting factors involved. Thus, experimentation in this scenario can be considered as an iterative process. Although it will provide a result, such methods are neither time nor cost effective. But the design of experiments is a scientific approach to effectively plan and perform experiments using statistics. In such designs, the combination of each factor at every level is studied to determine the combination that would yield the best result.

The design of experiments (DOE) is a powerful analysis tool for modeling and analyzing the influence of control factors on performance output. The most important stage in the DOE lies in the selection of the control factors. Therefore, a large number of factors are initially included so that non- significant variables can be identified at the earliest opportunity. A literature review on the erosion behavior of plasma sprayed coatings reveals that parameters viz., impact velocity, impingement angle, erodent size and erodent temperature largely influence the erosion rate. The impact of these four parameters on coating erosion is therefore studied in this work using Taguchi's L_9 (3^4) orthogonal array design. The control factors and their selected levels for erosion test are given in table 3.2.

Table 3.2 Control factors and their selected levels

Control factors	Levels			
	1	**2**	**3**	**Units**
A: Impact velocity	33	47	57	m/sec
B: Impingement Angle	30	60	90	mm
C: Erodent size	50	100	200	µm
D: Erodent Temperature	30	50	75	Degree

In a conventional full factorial design, it would normally require 3^4 = 81 runs to study five parameters each at four levels, whereas Taguchi's factorial experiment approach reduces it to only 9 runs, offering a great advantage in terms of experimental time and cost. The experimental observations are further transformed into signal-to-noise (S/N) ratios.

'Smaller the better' characteristic: $$\frac{S}{N} = -10\log\frac{1}{n}\left(\sum y^2\right) \qquad (3.1)$$

'Nominal the better' characteristics: $$\frac{S}{N} = -10\log\frac{1}{n}\left(\sum\frac{\bar{Y}}{S_Y^2}\right) \qquad (3.2)$$

'Larger the better' characteristics: $$\frac{S}{N} = -10\log\frac{1}{n}\left(\sum\frac{1}{y^2}\right) \qquad (3.3)$$

There are several S/N ratios available depending on the type of characteristics as given by the Eqns. 3.1, 3.2 and 3.3. Here, 'n' is the number of observations and 'y' is the observed data. The S/N ratio for minimum erosion rate comes under 'smaller the better' characteristic, which can be calculated as logarithmic transformation of the loss function by using Eq. (3.1).

Neural Network Analysis

Erosion wear process is considered as a non-linear problem with respect to its variables: either materials or operating conditions. As already mentioned, to obtain minimum wear rate, appropriate combinations of operating parameters have to be planned so as to study their interrelated effects and to predict the wear response under different operational conditions. To this end, a systematic analysis using another novel technique namely artificial neural network (ANN) is implemented in this work. ANN is a technique inspired by the biological neural system and has already been used to solve a wide variety of problems in diverse fields. It was developed to simulate the strong learning, clustering and reasoning capacity of biological neurons. With a strong learning capability and use of parallel computation and nonlinear mapping, neural networks can be successfully applied for identifying several nonlinear systems and control problems. The multiple layered ANN is the most extensively applied neural network for engineering materials like polymeric composites, but is seldom utilized in research in the field of wear-resistant coatings. The back propagation of ANNs can be used to train such multiple layered feed-forward networks with differential transfer functions to develop a functional model. Using a well-trained ANN model, one can estimate predictive performance, pattern association and pattern classification. As aforementioned, the erosion process is a complicated phenomenon lacking adequate mathematical description and therefore, a powerful method that combines the ANN technique and the Taguchi’s design is proposed in this study for better analysis and prediction of erosion performance of coatings. This proposed approach not only yields a sufficient understanding of the effects of process parameters, but also produces an optimal parameter setting to ensure that the coatings exhibit the best performance characteristics.

ANN is a technique that involves database training to predict input-output evolutions. Basically this technology is suitable for some complex, non-linear and multi-dimensional problems because it is able to imitate the learning capability of human beings. This means the network can learn directly from the examples without any prior formulae about the nature of the problem and generalize by itself some knowledge, which could be applied for new cases. A neural network is a system composed of many cross-linked simple processing units called neurons. The network generally consists of three parts connected in series: input layer, hidden layer and output layer. Experimental result sets are used to train the ANN in order to understand the input-output correlations. The database is divided into three categories, namely: (i) a validation category, which is required to define the ANN architecture and adjust the number of neurons for each layer. (ii) a training category, which is exclusively used to adjust the network weights and (iii) a test category, which corresponds to the set that validates the results of the training protocol. The coarse information is accepted by the input layer and processed in the hidden layer. Finally the results are exported via the output layer.

Chapter Summary

This chapter has provided:

1. The description of the materials used in the experiments
2. The details of fabrication of the coatings
3. The description of solid particle erosion wear test
4. An explanation of the Taguchi experimental design and neural computation

The next chapter presents the results and analysis of erosion tests conducted on the alumina coatings.

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Chapter 4
Results and Discussion

Chapter 4

RESULTS AND DISCUSSION:
EROSION RESPONSE OF ALUMINA COATINGS

Erosion wear characteristics of plasma sprayed 'alumina' coatings have been investigated in this study following a plan of experiments based on the Taguchi technique which is used to acquire the erosion test data in a controlled way. This chapter reports the wear rates obtained from these erosion trials and presents a critical analysis of the test results. Further, erosion rate predictions following an ANN approach for different test conditions are presented. A correlation among various control factors influencing the erosion rate has also been proposed for predictive purpose. Possible wear mechanisms are identified from the scanning electron microscopy of the eroded surfaces.

Erosion Test Results and Taguchi Analysis

In Table 3.6, each of the columns, from second to fifth, represents a test parameter, whereas a row stands for a treatment or test condition, which is nothing but a combination of the parameter levels. The plan of the experiments is as follows: the second column is assigned to impact velocity (A), the third column to impingement angle (B), the fourth column to erodent size (C) and the fifth column to erodent temperature (D) respectively to estimate the erosion rate (Er).

The test results for the erosion trials are given in table 4.1, in which the sixth column gives the erosion rate. Each data point (value of erosion rate) in this column is in fact the average of two replications. The last column represents S/N ratio of the erosion rate for each test run. From table 4.1, the overall mean for the S/N ratio of erosion rate is found to be -28.043 db. The analysis is made using the popular software *MINITAB-14* specifically used for design-of-experiment applications. Before any attempt is made to use this simple model as a predictor, the measures of performances must be considered. The response table (S/N ratio) for erosion rate is given as table 4.2 and the effects of control factors on erosion rate are shown in figure 4.1.

Test Run	Impact velocity A (m/sec)	Impingement angle B (degree)	Erodent size C (micron)	Erodent temperature D (Deg. cent.)	Erosion rate Er (Mg/kg)	S/N Ratio (dB)
1	33(1)	30(1)	50(1)	30(1)	19.537	25.8172
2	33(1)	60(2)	100(2)	50(2)	21.059	26.4688
3	33(1)	90(3)	200(3)	75(3)	22.586	27.0768
4	47(2)	30(1)	100(2)	75(3)	22.626	27.0922
5	47(2)	60(2)	200(3)	30(1)	23.485	27.4158
6	47(2)	90(3)	50(1)	50(2)	23.254	27.3300
7	57(3)	30(1)	200(3)	50(2)	25.165	28.0159
8	57(3)	60(2)	50(1)	75(3)	25.956	28.2848
9	57(3)	90(3)	100(2)	30(1)	25.475	28.1223

Table 4.1 L_9 Orthogonal Array and Erosion Test Results along with S/N Ratios

Level	A	B	C	D
1	-26.45	-26.98	-27.14	-27.12
2	-27.28	-27.39	-27.23	-27.27
3	-28.14	-27.51	-27.50	-27.48
Delta	1.69	0.53	0.36	0.37
Rank	1	2	4	3

Table 4.2 S/N ratio response table for erosion rate

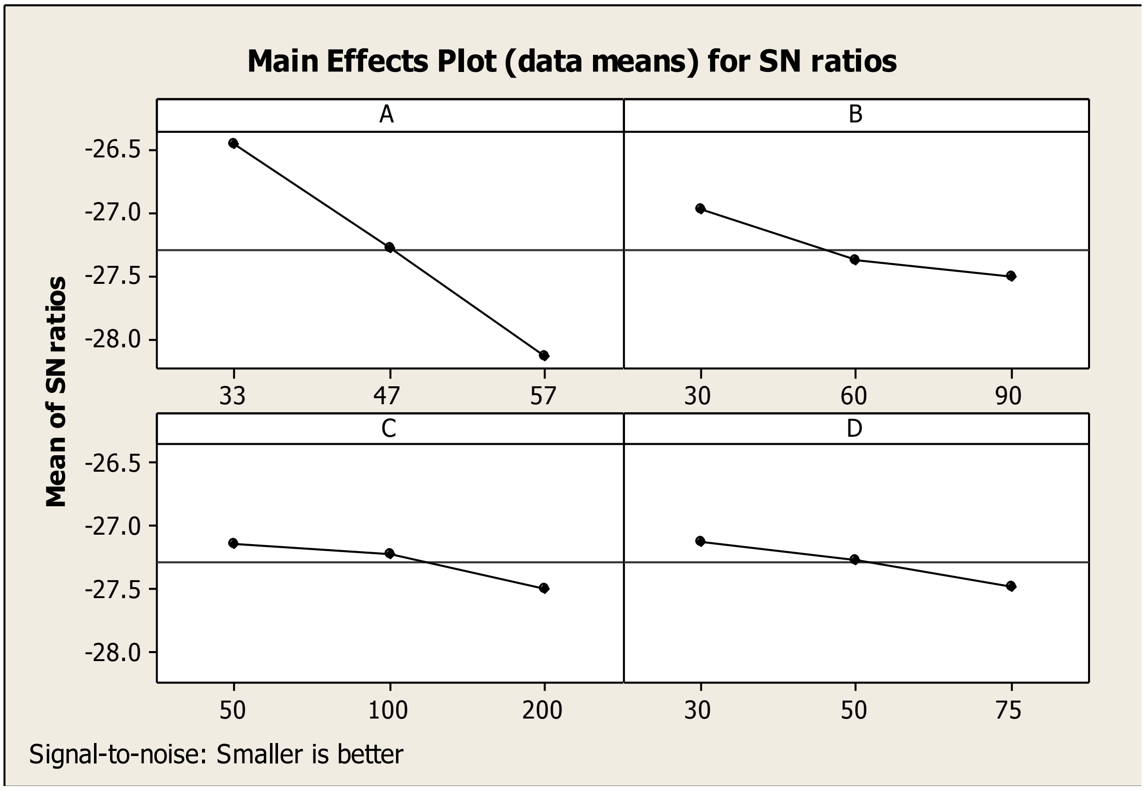

Figure 4.1 Effect of control factors on erosion rate

It is evident from the figure and the response table, that as far as the erosion rate is concerned, the factor combination A_1, B_1, C_2 and D_1 will give the minimum erosion rate. Further, table 4.2 indicates the hierarchical-order of the control factors as impact velocity (A), impingement angle (B), erodent size (C) and erodent temperature (D) in decreasing order according to their significance on the erosion rate. It can also be concluded that the erodent temperature (D) has negligible effect on the wear rate.

Analysis and Prediction of Erosion Response using ANN

As mentioned earlier, artificial neural network (ANN) is a technique that involves database training to predict input-output evolutions. In this attempt to simulate the erosion wear process and to predict the erosion rate under different operating conditions for alumina coatings, four input parameters are taken, each of which is characterized by one neuron in the input layer of the ANN structure. Experimental result sets are used to train the ANN in order to understand the input-output correlations. The input variables are normalized so as to lie in the same range of 0-1. The outer layer of the network has only one neuron to represent erosion wear rate. The neural network is constructed, taking the experimental database generated during the erosion trials on the coatings. About 75% of data is used for training, whereas 25% is used for testing.

Different ANN structures with varying number of neurons in the hidden layer are tested at constant cycles, learning rate, error tolerance, momentum parameter, noise factor and slope parameter. Based on least error criterion, one structure, shown in table 4.3, is selected for training of the input-output data. The optimized three-layer neural network used in this simulation is shown in figure 4.2. A software package NEURALNET for neural computing based on back propagation algorithm is used as the prediction tool for erosion wear rate of the coatings under various test conditions.

Input Parameters for Training	**Values**
Error tolerance	**0.001**
Learning rate (ß)	**0.002**
Momentum parameter(α)	**0.002**
Noise factor (NF)	**0.001**
Number of epochs	**1000,000**
Slope parameter (£)	**0.6**
Number of hidden layer neurons (H)	**10**
Number of input layer neurons (I)	**4**
Number of output layer neuron (O)	**1**

Table 4.3 Input parameters selected for training

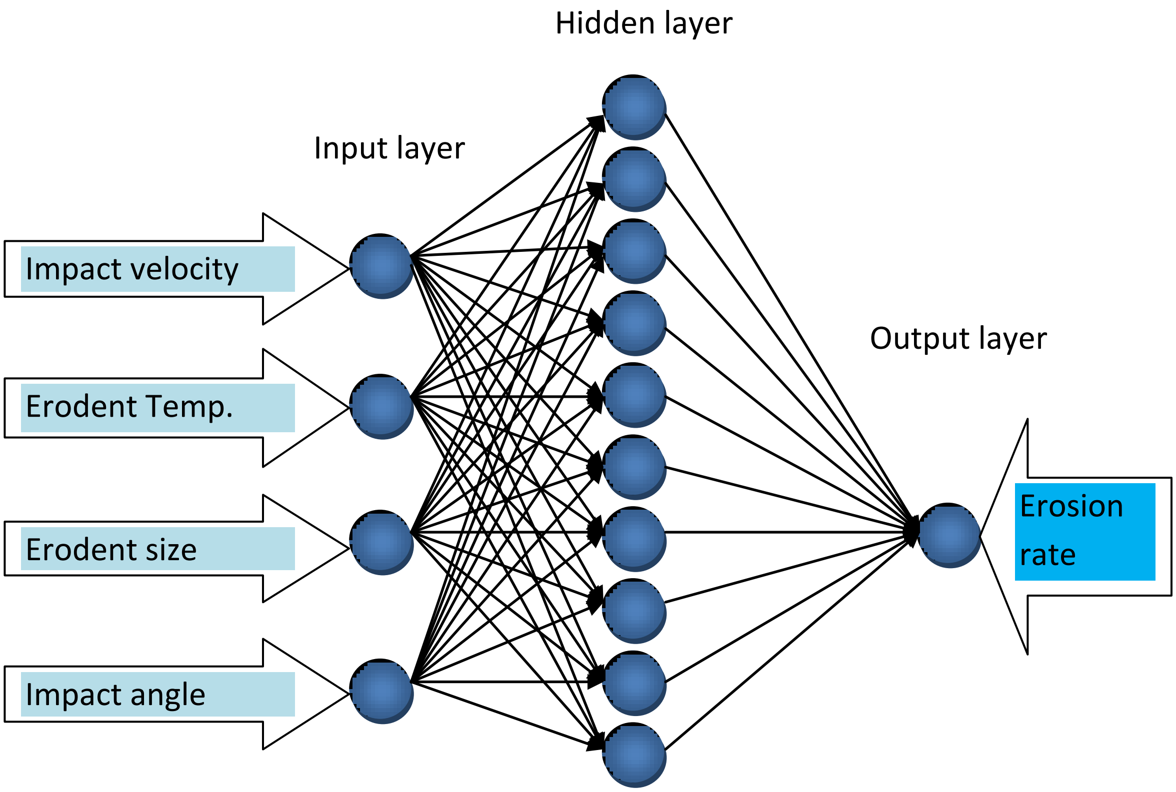

Figure 4.2 The three-layer neural network

Expt no.	Experimental value(mg/kg)	ANN predicted value(mg/kg)	Error(%)
1	19.537	19.58	0.22
2	21.059	20.838	1.04
3	22.586	22.451	0.59
4	22.686	23.079	2
5	23.485	23.59	0.44
6	23.254	23.494	1.03
7	25.165	25.12	0.17
8	25.956	25.219	2.83
9	25.475	25.274	0.78

Table 4.4 Percentage error between experimental result and ANN prediction

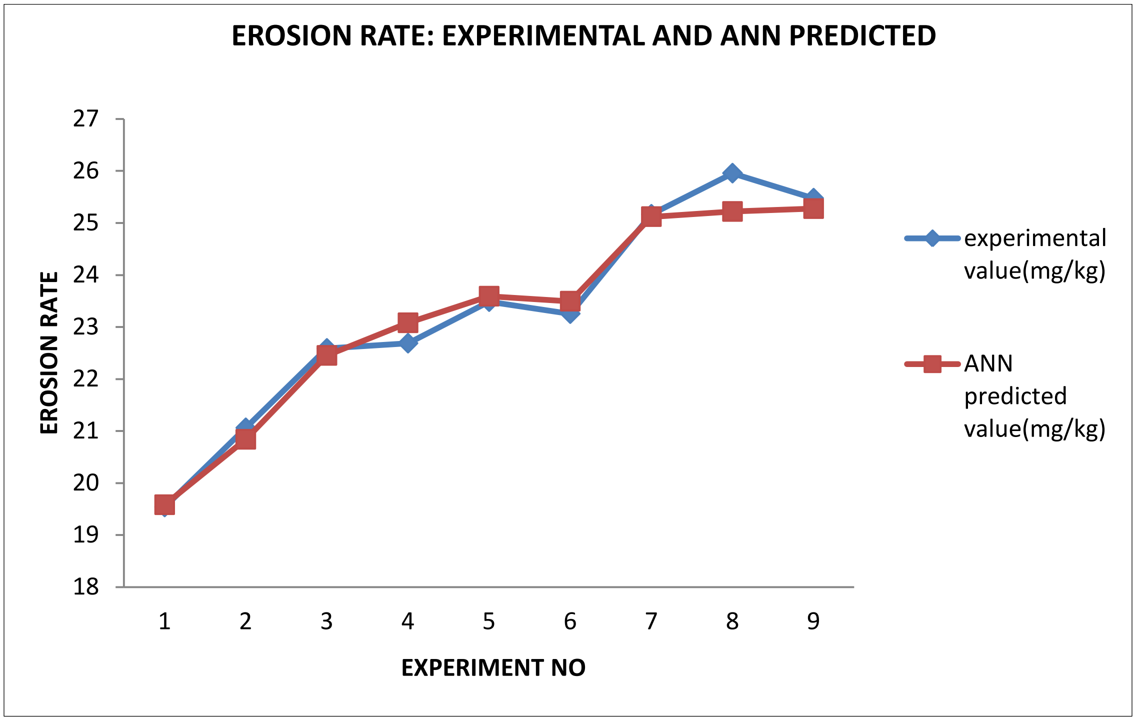

Fig 4.3 Comparison between the experimental results and ANN predictions

The ANN predictive results of erosion wear rate for all the 9 test conditions are shown and compared with the experimental values along with the associated percentage errors in table 4.4. It is observed that the errors lie in the range of 0-2% which establishes the validity of the neural computation.

A well-trained ANN is expected to be very helpful for the analysis of erosion wear characteristics of any given coating and permits to study quantitatively the effect of each of the considered input parameters on the wear rate. The range of any chosen parameter can be beyond the actual experimental limits, thus offering the possibility to use the generalization property of ANN in a large parameter space. In the present investigation, this possibility has been explored by selecting the most significant factor i.e. the impact velocity in a range from 25-65 m/sec. Sets of predictions for erosion rate of coatings at different impact velocities are evolved and this predicted evolution is illustrated in table 4.5 and figure 4.4.

<u>Erosion rate vs. Impact velocity</u> (at different impingement angles)

Erodent size: 100 micron, Erodent temperature: 75 ^{0}C

Impact velocity (m/sec)	Erosion Rate (mg/kg) at impingement angle(in degree)		
	30	**60**	**90**
25	19.306	19.914	20.451
33	20.641	21.267	21.806
47	23.079	23.736	24.274
57	24.885	25.561	25.097
65	26.357	27.047	27.582

Table 4.5 Predicted evolution of erosion rate with impact velocity & impingement angle

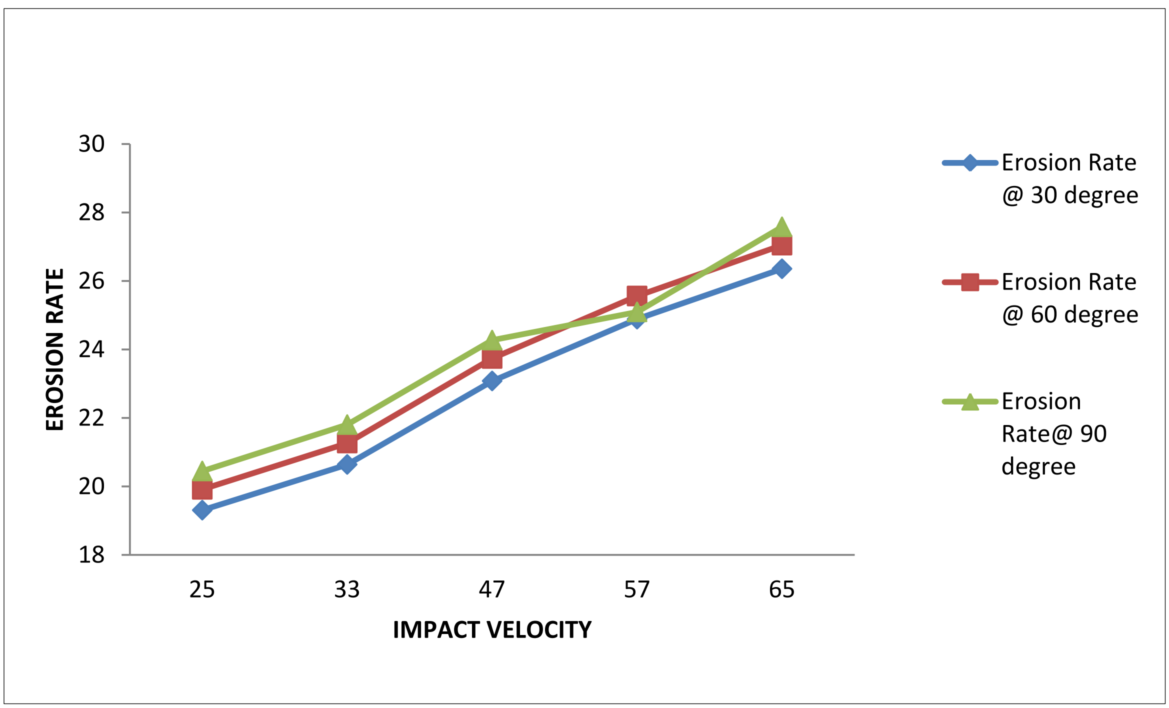

Figure 4.4 Predicted evolution of erosion rate with impact velocity & impingement angle

Similarly, sets of predictions for erosion rate of coatings at different impact velocities at different angles are evolved and this predicted evolutions are illustrated in tables 4.6, 4.7 and 4.8 and fin corresponding figures 4.5, 4.6 and 4.7 respectively.

<u>Erosion rate vs. Impact velocity</u> (at different erodent temperature)

Erodent size: 100 micron, impingement angle: 90 degree

impact velocity(m/sec)	**Erosion Rate(mg/kg) For diff. erodent size(micron)**		
	30	**50**	**75**
25	19.684	20.029	20.451
33	21.017	21.373	21.806
47	23.459	23.831	24.274
57	25.274	25.652	25.097
65	26.758	27.139	27.582

Table 4.6 Predicted evolution of erosion rate with impact velocity & erodent temp.

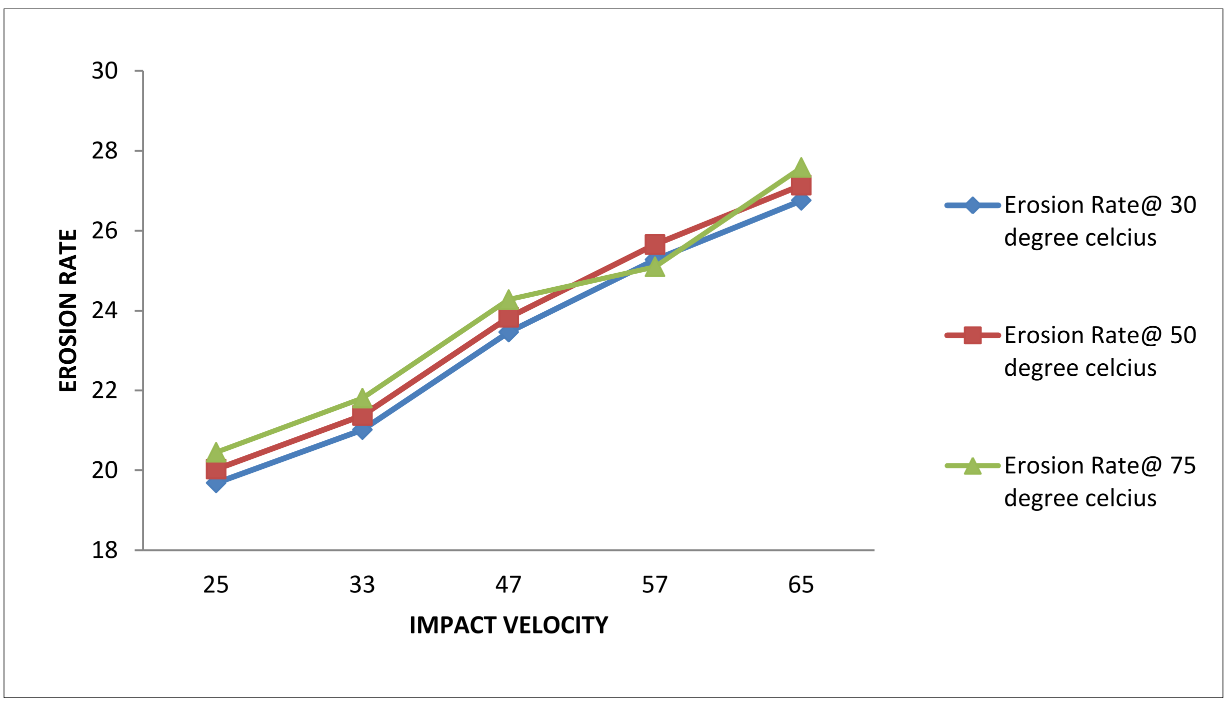

Figure 4.5 Predicted evolution of erosion rate with impact velocity & erodent temp.

<u>Erosion rate vs. Impingement angle</u> (different impact velocities)

Erodent temperature : 30 degree Celsius
Erodent size : 100 micron

impingement angle(degree)	**Impact velocity(m/sec)**		
	33	**47**	**57**
15	19.547	21.935	23.798
30	19.876	22.306	24.116
45	20.189	22.633	24.453
60	20.484	22.935	24.759
75	20.76	23.21	25.033
90	21.017	23.459	25.274

Table 4.7 Predicted evolution of erosion rate with impact angle & impact velocity

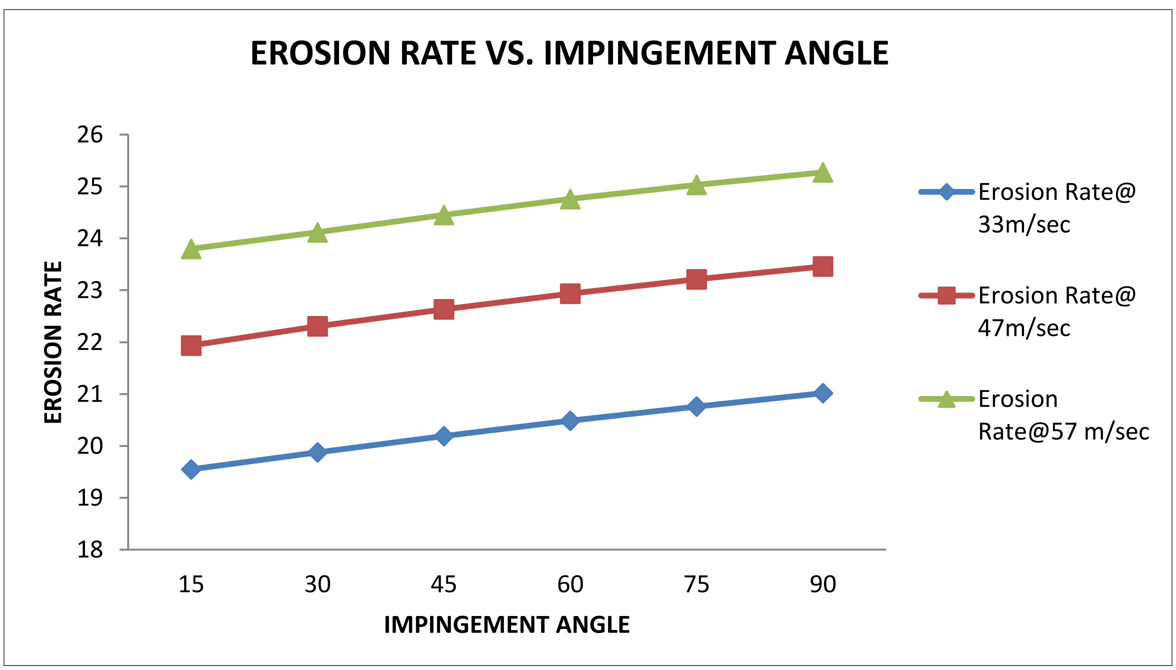

Figure 4.6 Predicted evolution of erosion rate with impact angle and impact velocity

Erosion rate vs. impingement angle (at different erodent temperatures)

Impact velocity: 57 m/sec
Erodent size: 100 micron

Impingement angle (degree)	Erodent temp. (degree Celsius)		
	30	**50**	**75**
15	23.748	24.092	24.498
30	24.116	24.469	24.885
45	24.453	24.815	25.24
60	24.759	25.127	25.561
75	25.033	25.407	25.846
90	25	25.652	26.097

Table 4.8 Predicted evolution of erosion rate with impact angle and erodent temp.

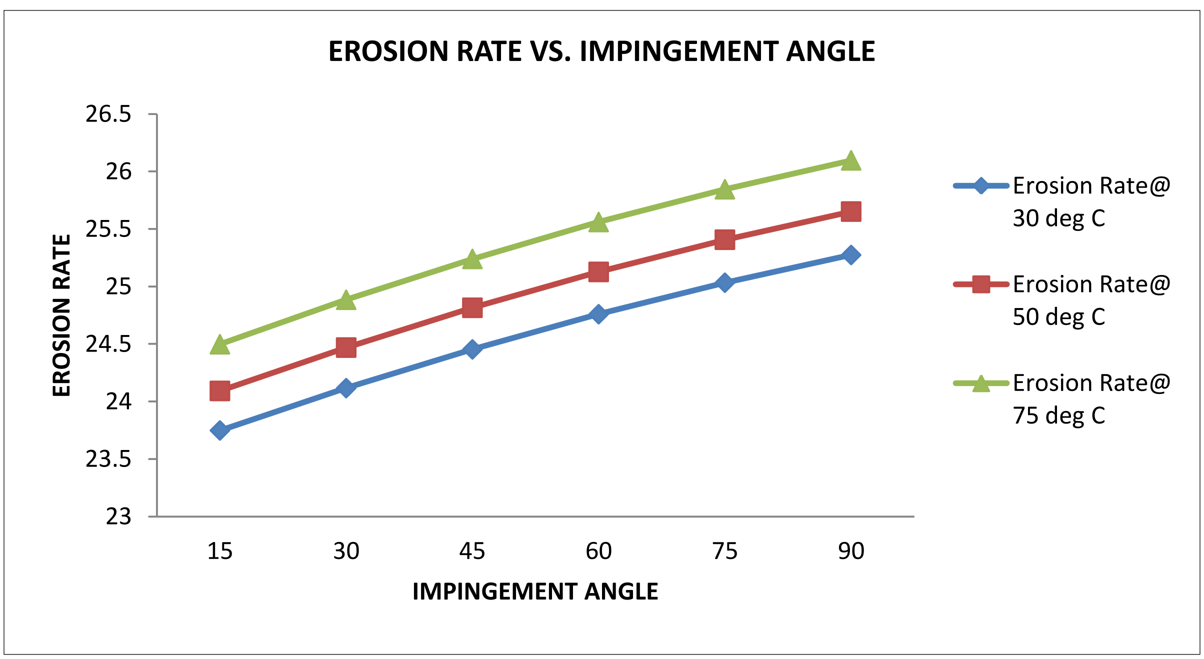

Figure 4.7 Predicted evolution of erosion rate with impact angle and erodent temp.

It is interesting to see that the erosion rate presents an exponential type evolution with the impact velocity. As the velocity of impact of the erodent increases, the kinetic energy carried by it also increases. This causes transfer of greater amount of energy to the target coating surface upon impact and leads to higher material loss due to erosion. Erosion rate (Er) depends on velocity (V) by a power law, given as $Er = kV^n$, where k is a material constant. However, the exponent 'n' is reported to be material independent and is governed by test conditions including particle characteristics and the erosion test apparatus.

Functional coatings have to fulfill various requirements when employed in tribological applications. The wear rate is one of the requirements as it is directly related to the service life period of the coatings. In order to achieve certain degree of erosion wear resistance accurately and repeatedly, the influence parameters of the process have to be controlled accordingly. Neural computation can be used as a predictive tool in such a case to process very large data related to a real time erosive situation and to simulate any desired parameter in a space larger than the domain of experimentation.

Chapter Summary

This chapter has provided a critical analysis on the solid particle erosion characteristics of alumina coatings using Taguchi experimental design.. The research presented in this chapter further illustrates that the use of a neural network model to simulate experiments with parametric design strategy is effective, efficient and helps to predict the solid particle erosion response of the coatings under different test conditions within and beyond the experimental domain. The predicted and the experimental values of erosion wear rate exhibit good agreement and validate the remarkable prediction capability of a well-trained neural network for this kind of processes.

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Chapter 5
Conclusions

Chapter 5

Conclusions

This investigation on solid particle erosion wear response of plasma sprayed alumina coatings has led to the following specific conclusions:

1. Commercially available alumina is eminently coatable on mild steel substrates employing atmospheric plasma spraying technique. Such coatings possess desirable coating characteristics.

2. Solid particle erosion characteristics of these coatings have been successfully analyzed using Taguchi experimental design. Significant control factors affecting the erosion rate have been identified through successful implementation of this technique. Impact velocity, impingement angle and erodent size in declining sequence are found to be significant for minimizing the erosion rate of these coatings. Erodent temperature is identified as the least influencing control factor for erosion rate.

3. All the coatings in this investigation exhibit brittle erosion response with the peak erosion occurring at normal impact (i.e. at 90^0 angle of impingement).

4. The use of an artificial neural network model to simulate experiments with parametric design strategy is effective, efficient and helps to predict the solid particle erosion response of such coatings under different test conditions within and beyond the experimental domain.

The experimental results support the potential of alumina to be used as a wear resistant coating material for deposition on mild steel substrates.

Recommendation for Potential Applications

The use of alumina coatings is suggested in structural applications such as electric towers and engineering trusses in deserts and mining sites. These coatings can also be recommended for engineering applications such as pipelines and valves carrying particulate matters, transport tubes carrying abrasive materials in an air stream, coal bends carrying pulverized coal, rocket motors trail nozzle, gun barrel, compressor, turbine and exhaust fan blades, burner nozzle, reheater, super heater and economizer tube banks, conveyer belt rollers etc..

Scope for Future Work

The present research work leaves a wide scope for future investigators to explore many other aspects of such plasma sprayed composite coatings. Some recommendations for future research include:

- The possible use of ceramic/metallic powders other than alumina
- Study on the response of these coatings to other wear modes such as sliding and abrasion
- Study on thermal stability of these coatings for high temperature applications
- Cost analysis of these coatings to assess their economic viability in industrial applications

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